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CHARACTERIZATION OF A THERMOPLASTIC POLYIMIDESULFONE

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INTRODUCTION

A polymer system which possessed the thermoplastic processability similar to polysulfones and the solvent resistance of polyimides was recently developed at the NASA-Langley Research Center. The synthesis and early characterization of this novel polyimidesulfone (PISO₂) has already been reported.¹ That study revealed the polymer was relatively economical and easy to synthesize and could be processed as a thermoplastic. The combination of good mechanical properties and thermal stability make this resin attractive for use in high temperature structural applications.

Problems, however, exist with the PISO₂ adhesive in terms of solvent retention and foaming. Foaming can cause voids when producing large area bonds or bulky composite parts and result in reduced structural properties. The empirical development of processing cycles often directly follows the synthesis of a new adhesive. Extensive chemical characterization of the polymer system would probably result in a better understanding of the adhesive and help solve potential processing problems. The objective of the present study was to conduct a detailed chemical characterization of the PISO₂ adhesive and corresponding model compounds in an effort to optimize processing cycles.

EXPERIMENTAL

Polymer Preparation. Monomers used to synthesize the polyimidesulfone (PISO₂) were 3,3',4,4'-benzophenonetetracarboxylic dianhydride (BTDA) and 3,3'-diaminodiphenylsulfone (3,3'-DDSO₂).

The BTDA was received from King Mar Laboratories*, San Diego, CA and used after sublimation at 210°C, m.p. 222-224°C. The 3,3'-DDS₂ was obtained from FIC Corporation, San Francisco, CA and was dried overnight in a vacuum oven at 80°C before use, m.p. 168-170°C. Polymerizations were conducted in reagent grade bis(2-methoxyethyl) ether, or diglyme, as received from Fluka Chemical Corp., Hauppauge, NY, b.p. 162°C. Starting material purity was verified by differential thermal analysis (DTA) and elemental analysis.

Reactions were conducted in a closed vessel at 20-25°C at 15-20% solids by weight in diglyme. A typical small scale synthesis for characterization studies was as follows: 6.50 g (0.02 mole) of BTDA was added in small portions to a solution of 5.01 g (0.02 mole) 3,3'-DDS₂ in 64.7 g diglyme. The solution was allowed to stir for several hours after all of the BTDA had dissolved. The precursor polyamide-acid in solution was stored in a freezer prior to use.

Model Compounds Preparation. Model compounds of the PISO₂ system were prepared under the same conditions as the polymer except that the monofunctional phthalic anhydride was used in place of the bifunctional BTDA. Phthalic anhydride was received from Eastman Kodak Co., Rochester, NY and used after sublimation at 120°C, m.p. 130-132°C. The model amide-acid was synthesized at room temperature at 15% solids by weight in diglyme.

Preparation of Model Amide-Acid. 3,3'-bisphthalamide acid diphenylsulfone (I)

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A typical small scale synthesis was as follows: 1.48 g (0.01 mole) phthalic anhydride was added in small portions to a solution of 1.24 g (0.005 mole) 3,3'-DDSO₂ in 14.65 g diglyme. The solution was allowed to stir several hours after all of the phthalic anhydride had dissolved. The product was precipitated into water, recovered, washed with water, and dried.

Anal. Calcd. for C₂₈H₂₀N₂O₈S: C, 61.76; H, 3.70; N, 5.14; O, 23.51; S, 5.89. Found: C, 61.68; H, 3.79; N, 5.13; O, 23.61; S, 5.98.

Thermal Imidization of Model Amide-Acid. 3,3'-bisphthalimide diphenylsulfone (II)

Compound I, 2.00 g, in a covered beaker, was placed in a forced air oven at 200°C for 0.5 hour. This heating yielded an analytically pure imide.

Anal. Calcd. for C₂₈H₁₆N₂O₆S: C, 66.14; H, 3.17; N, 5.51; O, 18.88; S, 6.30. Found: C, 66.20; H, 3.22; N, 5.53; O, 18.76; S, 6.40.

Powder Preparation. Both the polyamide-acid and model amide-acid solutions were precipitated by pouring into rapidly stirring distilled water. The beige solid products were suction filtered and allowed to air dry overnight. The polymer and model were then heated in a vacuum oven at 80°C overnight to remove residual solvent.

Characterization. Inherent viscosity measurements were conducted at 35°C on polyamide-acid solutions at 0.5 percent solids in N,N-dimethylacetamide. Glass transition temperatures (T_g) for all specimens were determined by thermomechanical analysis (TMA) on a DuPont 943 Analyzer in

static air at 5 or 10°C/min heat rise rates. Thermal analyses were also performed on powder and adhesive scrim cloth specimens by differential scanning calorimetry (DSC) in static air at 5°C/min using a DuPont 990 Thermal Analyzer system. Thermogravimetric analyses (TGA) were obtained in air on a Perkin-Elmer TGS-2 at a temperature program of 2.5°C/min. A Perkin-Elmer Model 297 Infrared Spectrophotometer was used to determine the IR spectra of the various materials dispersed in KBr pellets. Spectra were also obtained using a Nicolet Model 60SX Fourier Transform Infrared Spectrometer (FTIR) using a dynamic diffuse reflectance (DR) technique to monitor imidization while heating a sample at 9°C/min. Mass chromatograms were obtained on a Finnigan 3300 Gas Chromatograph/Mass Spectrometer equipped with a solid probe. Samples were programmed at 10°C/min for this analysis. Lap shear strength of adhesive bonds were obtained according to ASTM D-1002 using a Model II Instron Universal Testing Machine. Specimens were tested at room temperature and 204°C in a clam-shell, quartz-lamp oven and held at temperature for ten minutes prior to testing.

Preparation of Adhesive Scrim Cloth. Adhesive scrims were prepared by brush-coating a polyamide-acid solution (15% solids in diglyme) onto 112 E-glass cloth with an A-1100 finish (γ -aminopropylsilane). The glass cloth, which had a nominal thickness of 0.01 cm (0.004 in), was stretched tightly over a metal frame and oven-dried for 2 hr at 150°C prior to coating. Coatings of polymer solution were applied to build up a scrim thickness of approximately 0.023 cm (0.009 in). After each coating, the scrim was air dried at room temperature and then placed in a forced-air oven and staged according to the following cure schedule:

- (1) Room temperature (RT) \rightarrow 60°C, hold 10 min
- (2) 60°C \rightarrow 100°C, hold 1 hr
- (3) 100°C \rightarrow 150°C, hold 1 hr

Portions of the adhesive scrim were removed and one portion was staged an additional 0.5 hr at 175°C, another portion an additional 0.5 hr at 200°C, and the final portion an additional 6 hr at 250°C.

Flow Characterization. Adhesive flow characteristics were determined by rapidly heating a 1.27 cm (0.5 in) diameter scrim cloth specimen in a press equipped with an induction bonding unit² and holding for 3 min at temperature under a constant 1.38 MPa (200 psi) pressure. The degree of flow was determined by measuring the change in area of the scrim specimen using a planimeter. The discs were sandwiched between two Kapton films and a plate of aluminum alloy (bottom) and titanium alloy (top). This sandwich was located on a steel mesh acting as the heating element. A thermocouple was placed in the sandwich such that the temperature of the specimen could be measured. Two layers of quartz-felt insulating material covered both the top plate and steel plate to facilitate heat transfer from the bottom plate up and to minimize temperature fluctuations. This experiment was first performed on seven individual specimens of the 150°C pre-staged scrim cloth at press temperatures of 204°C (400°F) to 371°C (700°F) in increments of 28°C (50°F). The test was also performed on the three remaining adhesive scrims at a 343°C test temperature.

Adhesive Bonding. The adhesive scrim cloth was used to bond 0.127 cm (0.05 in) thick titanium adherends (Ti 6Al-4V). The four-fingered titanium

adherend panels were surface treated with a modified Pasa Jell 107* process. Treated adherends were primed immediately to protect the oxide surface with a thin coat of the 15% solids polyamide-acid solution and allowed to air dry. All primed adherends were staged initially in a forced-air oven for 10 min at 60°C, 1 hr at 100°C, and 1 hr at 150°C. Three sets of Ti adherends were staged additionally with the adhesive scrim cloth 0.5 hr at 175°C, 0.5 hr at 200°C, or 6 hr at 250°C respectively.

Single lap shear specimens were prepared by inserting the scrim cloth between the primed adherends using a 1.27 cm (0.50 in) overlap and bonded according to the following two cycles (ASTM D-1002). The first was based on a previously reported processing cycle.¹

Standard Cycle

- (1) Contact pressure, heating rate 5°C/min, RT + 325°C, apply 1.38 MPa (200 psi) at 280°C
- (2) Hold 15 min at 325°C
- (3) Cool under pressure to ~150°C and remove specimens from press.

Modified Cycle

Same as standard cycle except that pressure (200 psi) was applied at 200°C.

*Trade name for a titanium surface treatment available from Semco, Glendale, CA.

RESULTS AND DISCUSSION

Model Chemistry and Properties. Model compounds of the polyimidesulfone system were synthesized according to the reaction scheme in Figure 1. These compounds were formed under the same conditions as the polymer, but produced an easily characterized low molecular weight material that modeled a portion of the polymer repeat unit. The reaction was performed in diglyme at room temperature. Figure 2 shows an infrared spectrum of the model amide-acid powder. Characteristic bands are identified, particularly those related to amide and acid functional groups.

Thermal analyses performed on the model amide-acid powder revealed that the imidization reaction was complete at 200°C as shown by both the large DSC endotherm in Figure 3 and the first weight loss hump in the TGA in Figure 4. The initial weight loss was due to the loss of water during the conversion of amide-acid to imide. The second, sharper endotherm at 317-320°C in Figure 3 is due to the melting of the imide.

Figure 5 shows an infrared spectrum of the model imide powder after being imidized in an air oven at 200°C for 0.5 hour. Amide-acid bands that were previously observed are gone and replaced by five characteristic bands at 1778, 1718, 1375, 1110, and 718 cm^{-1} associated with imide ring vibrations.³ Heating the amide-acid to 200°C gave analytically pure imide.

Polymer Chemistry and Properties. The polyimidesulfone system was synthesized according to the reaction scheme shown in Figure 6. Diglyme was chosen as the solvent for this reaction because of monomer solubility and because it was a better solvent in associating strongly with DDSO_2 .⁴ Diglyme had been reported to yield polymers with high adhesive

strengths,^{1,5} and should be easily removed during cure so that moldings and laminates can be prepared free of voids.⁶ The first stage of the synthesis was conducted at room temperature, which was the optimum reaction temperature for producing high molecular weights in similar polyimide systems.⁷ The resulting polyimide precursor, the polyamide acid, was a deep amber color probably due to the formation of interchain charge transfer complexes.^{3,8} Polyamide-acid inherent viscosities ranged from 0.6 to 0.75 dl/g. The thermal imidization of the polyamide-acid should result in a linear, high molecular weight polyimidesulfone possessing adequate flow properties to be processed as a thermoplastic. Part of the thermoplasticity reportedly relates to the flexibility of the linear polyimide due to meta linkages in the aromatic diamine.¹

Thermal analyses were performed on the polyamide-acid powder. The first DSC run on an amide-acid sample shown in Figure 7, exhibited a large endotherm peaking at about 200°C, probably due to the imidization reaction. After heating to 340°C, the sample was cooled to room temperature. The second heat-up curve exhibited an inflection at 275°C, indicative of the T_g of the amorphous polymer. Figure 8 shows a TGA of the polyamide-acid powder. A gradual loss of weight is observed at about 175°C due to loss of water during imidization. From DSC and TGA data, the imidization reaction appears to occur in the temperature range of 150-225°C. The polyamide-acid imidized at a higher temperature and more gradually than the model amide-acid probably because of the decrease in mobility of the polymer.

Dynamic diffuse reflectance-FTIR was used to obtain the infrared spectra of opaque materials like the polyamide-acid powder and adhesive scrim cloth. This technique had been utilized by other NASA-Langley

researchers to monitor the cure of preregs and composites.^{9,10} Figures 9 and 10 show some representative infrared spectra from the analysis of a polyamide-acid powder sample. The first spectra at 43°C revealed bands due to the amide and acid portions of the polymer. The amide bands located at 3315 and 1535 cm^{-1} and the acid band at about 2600 cm^{-1} decreased in intensity as the sample was heated. Bands due to the imide ring also appeared and increased in intensity as the imidization reaction proceeded. After heating to 252°C the spectrum indicated a fully imidized structure with characteristic imide bands at 1780, 1720, 1360, and 720 cm^{-1} and no amide or acid bands. This was the same behavior observed on the model compounds. This work showed the imidization process to be complete by about 250°C.

The possibility of imidizing the polyamide-acid by holding for a longer period of time at a lower temperature was also investigated. A 0.0025 cm (0.001 in) thick polyamide-acid film was cured for 5 days at 150°C in a forced-air oven. According to the top TGA curve in Figure 11 and the top IR spectrum in Figure 12, the film sample was fully imidized. The same experiment performed on a polyamide-acid powder sample resulted in only partial imidization as evidenced by the bottom TGA curve in Figure 11 and the bottom IR spectrum in Figure 12. The polymer film was imidized to a greater extent than the powder after being thermally exposed the same. This experiment showed that films and powders may not imidize to the same degree under the same conditions and pointed to the need to characterize the material in the physical state in which it is to be used.

Characterization of Adhesive Scrim Cloth. In light of results obtained in characterizing the neat polymer, the adhesive scrim cloth was extensively characterized before processing. Mass spectral data was obtained on the

150°C pre-staged scrim cloth to identify any volatiles present. Figure 13 shows the mass chromatograms of water ($m/e = 18$) and diglyme as a function of temperature and indicates where each is evolving at 10^{-6} torr. The top trace shows water coming off between 140°C and 240°C. The other traces show the main fragments of diglyme ($m/e = 29, 45, 59, \text{ and } 89$), which evolved between 180°C and 240°C. The boiling point of diglyme at atmospheric pressure is 162°C. Evidently the solvent was physically bound to the adhesive, since such a high temperature was required to remove it under high vacuum. Foaming of the adhesive scrim cloth observed after staging at about 200°C was due to this residual solvent and water of imidization.

Some of the scrim cloth was further heat treated in order to decrease the volatile content. The TGA data in Figure 14 shows the difference in volatile content between the four pre-staged scrim cloth samples. The volatile content of the adhesive scrim cloth after the individual thermal staging were as follows: 150°C, 12%; 175°C, 9%; 200°C, 3%; and 250°C, < 1%. Diffuse reflectance infrared data on these specimens verified the difference was due to the loss of solvent and water of imidization. After staging the scrim cloth to an extreme of 250°C, the material was fully imidized and essentially solvent-free. T_g s obtained by TMA on the scrim cloth specimens and primed titanium surfaces before and after bonding are given in Table I. The before bonding values for the scrim cloth ranged from 158°C to 216°C and increased as the staging temperature was increased. The same trend was observed with the primed Ti surface values. After bonding at 325°C, the T_g s for the fractured lap shear specimens tested at RT were essentially constant. These values were lower than the 275°C T_g obtained for the PISO₂ powder by DSC. This behavior was also noted in an earlier

processing study on this adhesive.¹¹ An important point is that the apparent T_g s were well below the 275°C T_g of the neat resin. In previous work,^{1,11} pressure was usually applied during bonding at or near the T_g of the neat resin. The present work suggests that pressure should be applied at a lower temperature, perhaps around 200°C.

In order to assure a sufficient amount of adhesive flow to wet the bonding surfaces, flow measurements were made on 7 specimens of 150°C pre-staged scrim cloth at a series of exposure temperatures. The results of these measurements are displayed in Figure 15. A 100% flow value indicates a doubling in specimen area. The data shows a gradual increase in flow as the press temperature increased up to above 315°C. No additional flow was observed above 315°C. This data gave an indication of the minimum temperature required to produce the maximum amount of flow. This flow experiment was repeated at one test temperature on each of the four pre-staged scrim cloths. Figure 16 gives data on specimens heated at 343°C. The 175°C staged scrim sample flowed only one-third as much as the 150°C staged scrim. The scrim cloths staged at 200°C and 250°C showed essentially no flow. The volatile content of the scrim as shown by TGA in Figure 14 followed the same trend as the flow data, which suggests the two are related. If flow or wetting is a critical factor in adhesive bonding, then the lap shear data obtained with these specimens should exhibit the same trend as the flow data.

Lap Shear Bonding and Testing. The two processing cycles used to bond the adhesive specimens are portrayed in Figure 17. The standard cycle involved heating the specimens from RT to 325°C, applying pressure at 280°C, close to the T_g of the neat resin, holding at temperature 15 minutes, and

cooling under pressure to below the T_g before removing specimens from press. A modified cycle was also used which involved the same conditions except pressure was applied at 200°C, a temperature closer to the apparent T_g of the specimens. The 325°C bonding temperature was sufficient to yield the maximum flow. Initially, both bonding cycles were used to process the 150°C pre-staged scrim specimens. The RT lap shear values are presented in Figure 18 and represent the average of at least four specimens per condition. The lap shear strengths of the 150°C pre-staged specimens using the standard cycle were about 3500 psi. Values obtained using the modified cycle showed an 18% increase, a significant improvement. Thus, the modified cycle was utilized to process the remaining adhesive specimens.

Figure 19 gives lap shear results using the modified cycle to bond titanium adherends with the four different pre-treated scrim specimens. Measurements were made at RT and 204°C. The RT values for the 150°C and 175°C specimens were in the same range. Lap shear strengths for the 200°C and 250°C specimens were lower, but perhaps not as low as might be predicted considering the flow data alone. Thus, adhesive flow is evidently not the only contributing factor for successful bonds. The polyimidesulfone system can be processed as a thermoplastic because of respectable bonding properties even with low flow. If the decreased strengths obtained after extreme staging can be tolerated, volatiles should not be a problem during large area bonding.

SUMMARY

A fundamental characterization study was conducted on a polyimidesulfone adhesive. The significant results are summarized as follows: Detailed chemical characterization of model compounds as well as the polymer was useful in understanding adhesive behavior and performance. The importance of characterizing the polymer in the state used, not just relying on neat resin results, was recognized. Film and powder specimens given the same thermal exposure were not imidized to the same extent; fully cured neat resins and adhesive scrim cloths exhibited different T_g s; a polyamide-acid film was fully imidized by holding for 5 days at 150°C.

Thermomechanical analysis of the scrim cloth suggested that pressure should be applied during the bonding cycle at 200°C, close to the apparent T_g of the specimens. Mass spectrometry and infrared analysis of the adhesive scrim cloth showed that the foaming observed around 200°C was due to the evolution of residual diglyme solvent and water of imidization. A solvent free, fully imidized scrim cloth was obtained after an extreme staging of 6 hrs at 250°C.

Flow studies of 150°C pre-staged scrim cloth showed that 315°C was sufficient to produce maximum adhesive flow. Similar studies for a series of scrim specimens staged above 150°C showed decreased flow correlated with decreased volatile content.

Higher lap shear strengths were obtained using a modified cycle, where pressure was applied at the apparent T_g of the adhesive scrim, compared to the standard cycle. The best RT values, about 4200 psi, resulted using the 150°C and 175°C pretreated specimens. Lower strength values, 3100 psi and

2650 psi, were obtained with the 200°C and 250°C pretreated specimens, respectively. Adhesive properties were related to, but not totally dependent upon flow properties. A compromise should be reached on volatile content to optimize the bonding properties; too much volatiles causes excess foaming, too little yields little or no flow. An extensive characterization study of this nature leads to a better understanding of the chemistry of the adhesive and aids in optimizing the processing conditions.

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Table I. TMA results of adhesive scrim cloth specimens and titanium adherends.

PRETREATMENT ^A	GLASS TRANSITION TEMPERATURE, °C			
	BEFORE BONDING		AFTER BONDING ^B	
	ADHESIVE SCRIM CLOTH	PRIMED Ti SURFACE	SCRIM CLOTH FLASHING	BONDLINE Ti SURFACE
PRE-STAGE ONLY	158	165	248	232
1/2 HR. @ 175°C	163	178	248	226
1/2 HR. @ 200°C	210	218	248	229
6 HR. @ 250°C	216	232	254	235

^A ALL SPECIMENS WERE PREVIOUSLY STAGED 10 MIN. @ 60°C, 1 HR. @ 100°C, AND 1 HR. @ 150°C IN AIR.

^B LAP SHEAR SPECIMENS WERE BONDED AT 325°C AND 200 PSI, AND TESTED AT ROOM TEMP.

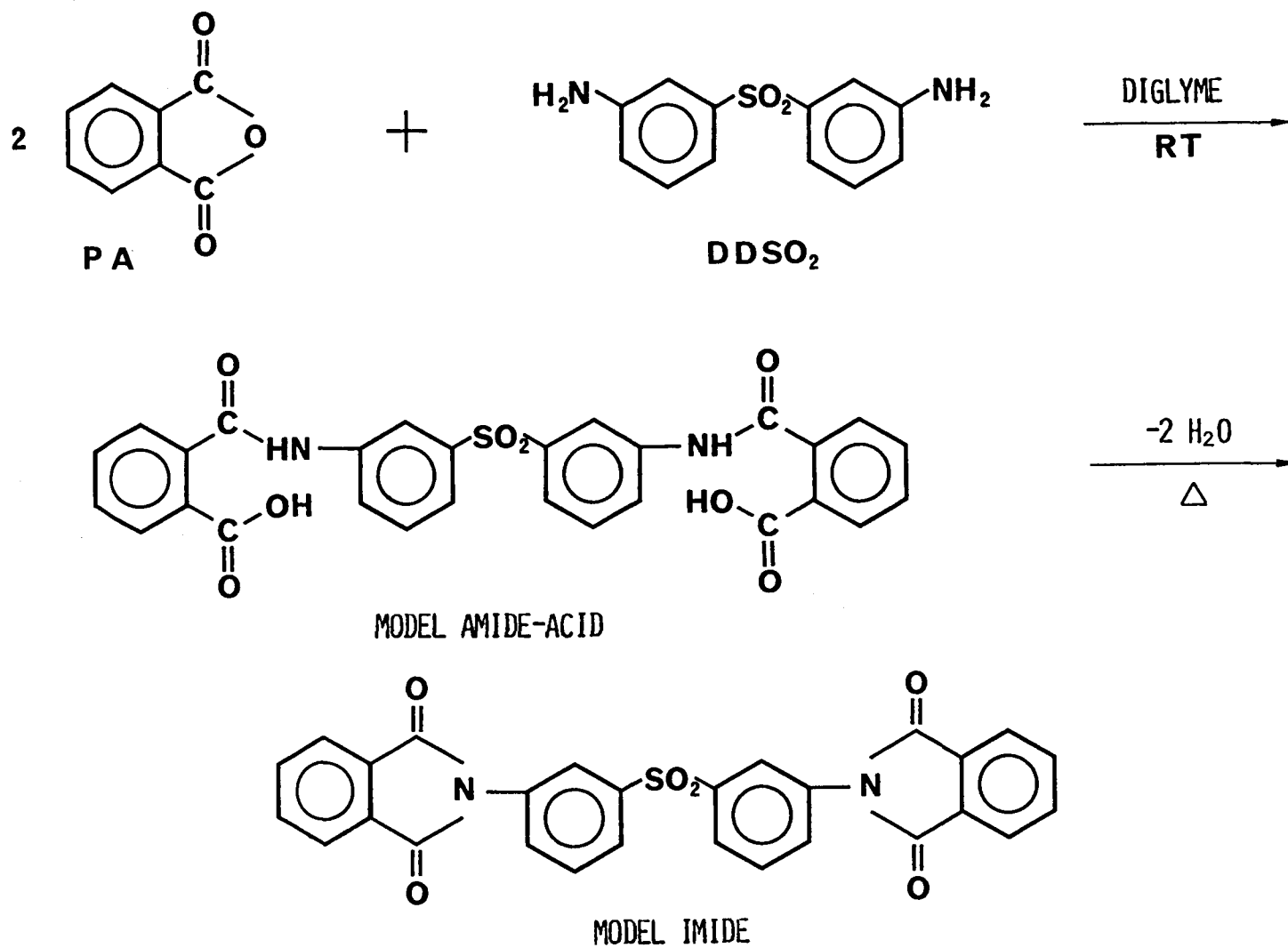


Figure 1. Reaction scheme of model compounds.

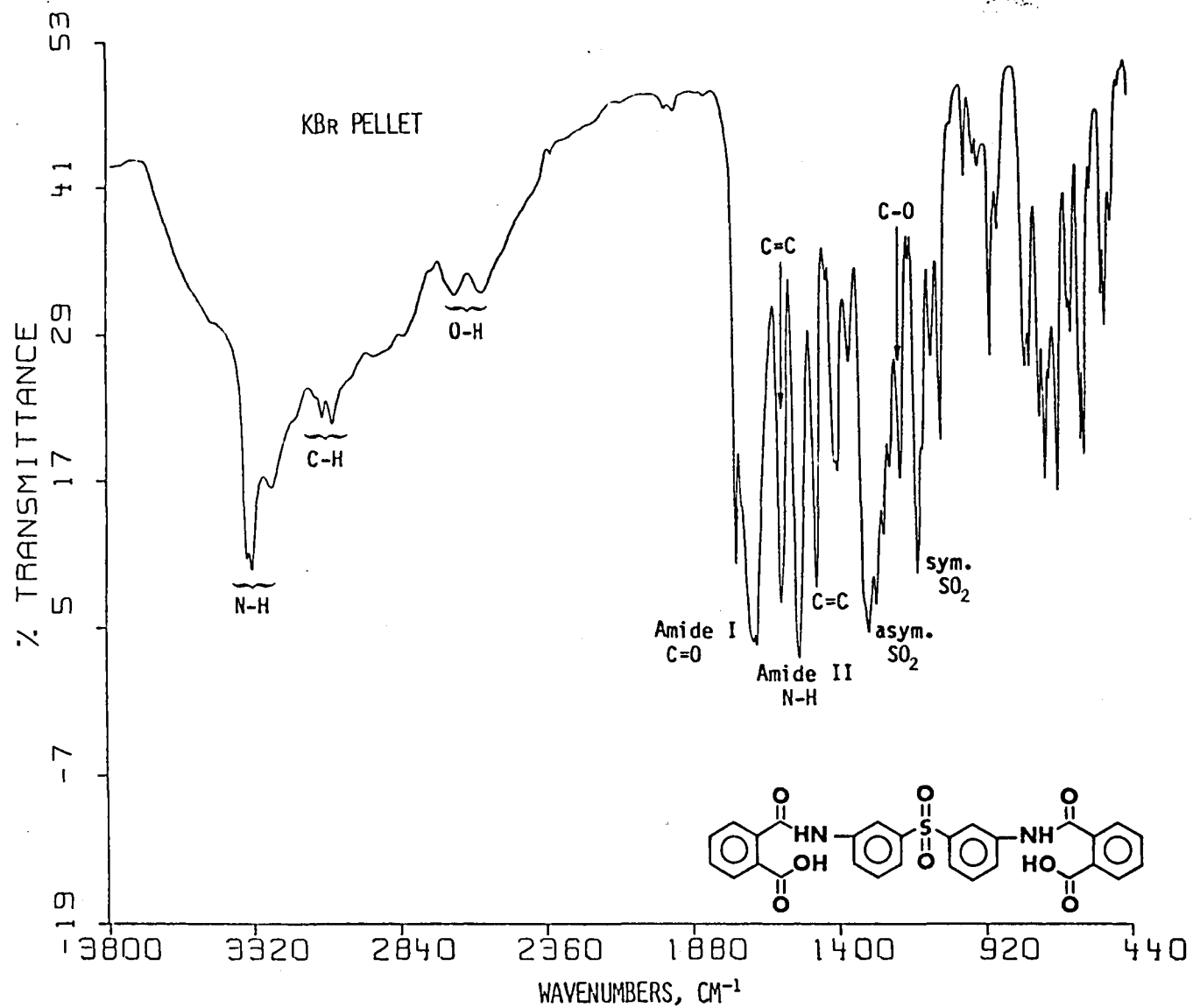


Figure 2. Infrared spectrum of model amide-acid.

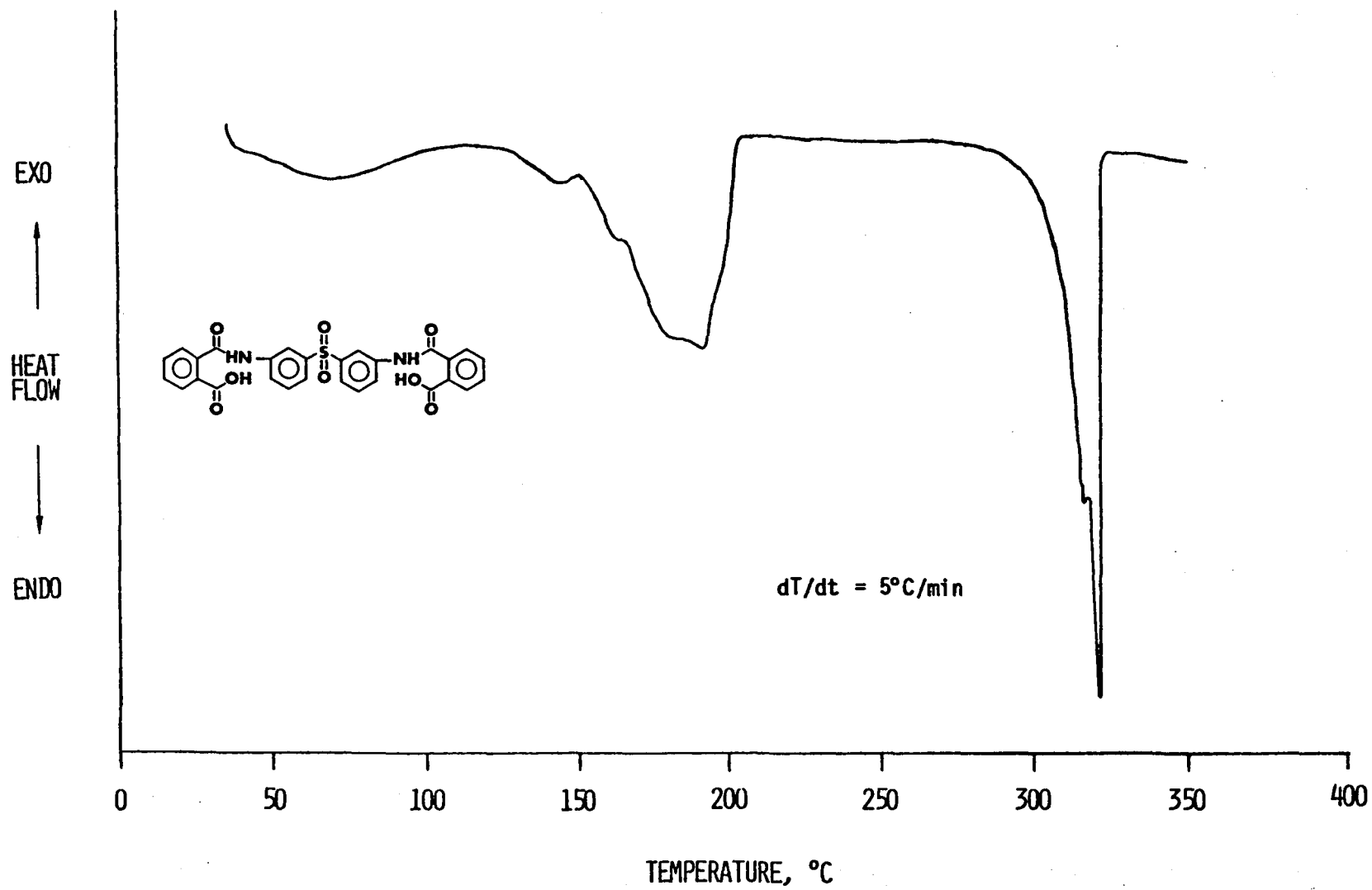


Figure 3. DSC of model amide-acid.

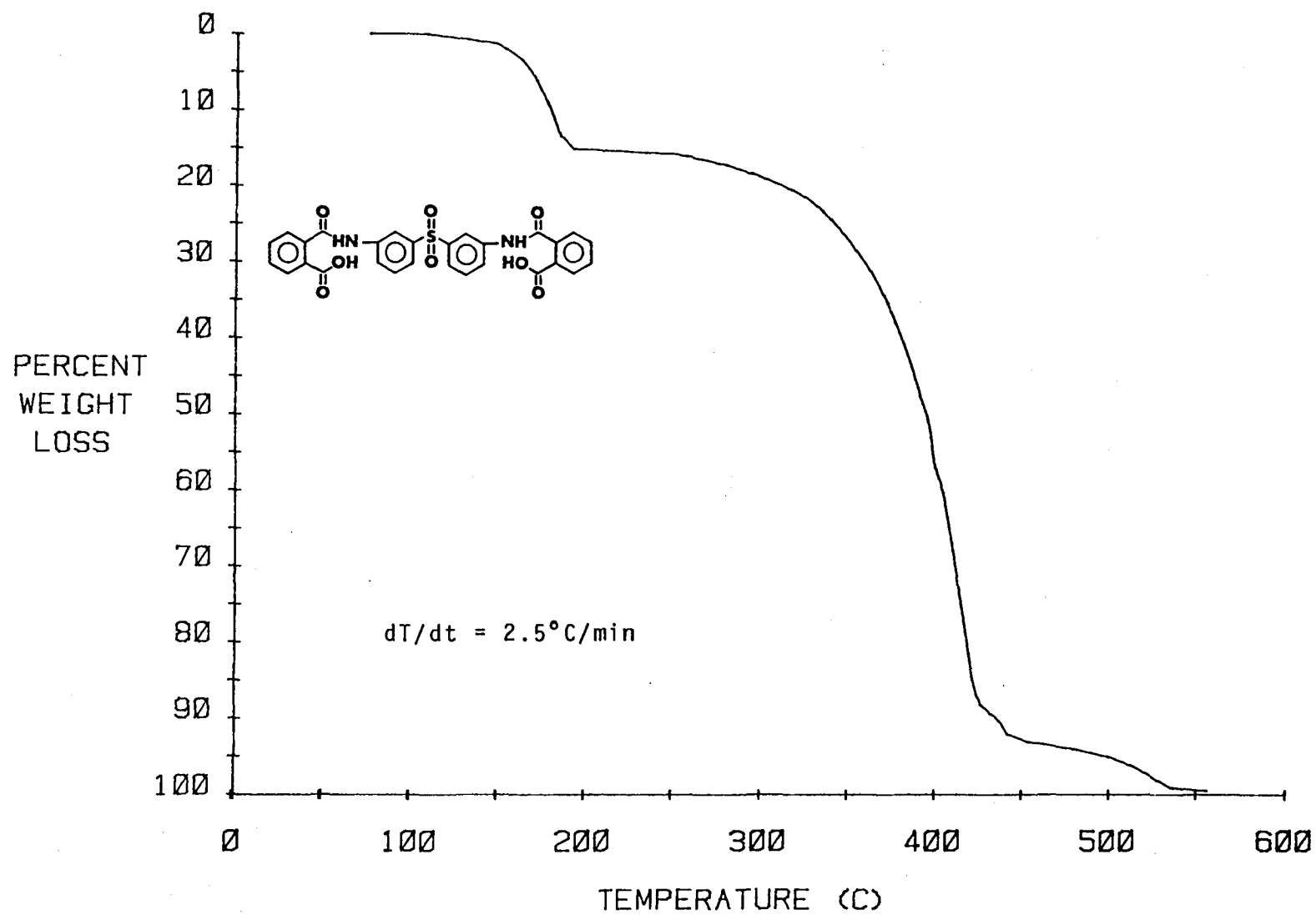


Figure 4. TGA of model amide-acid.

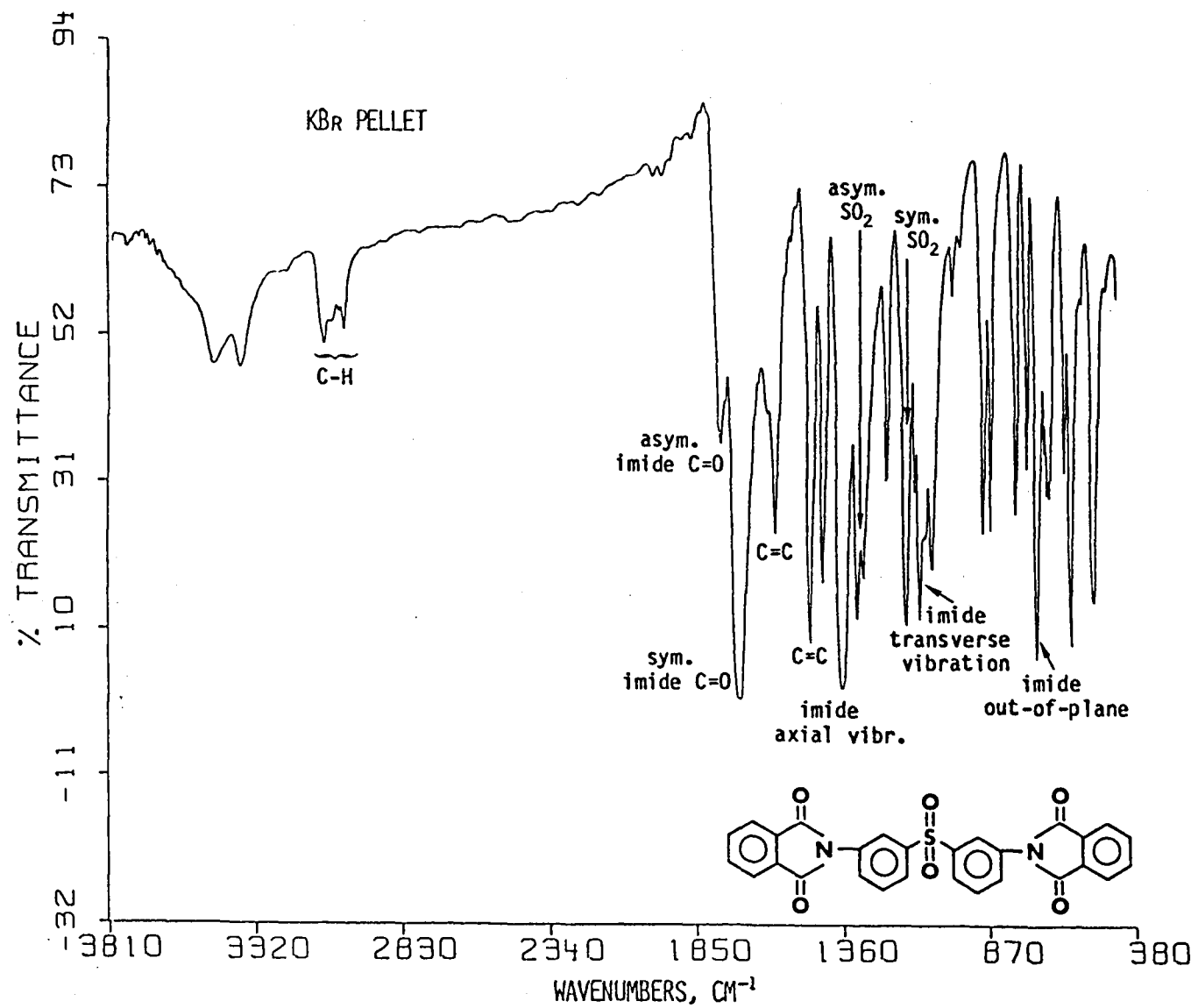


Figure 5. Infrared spectrum of model imide.

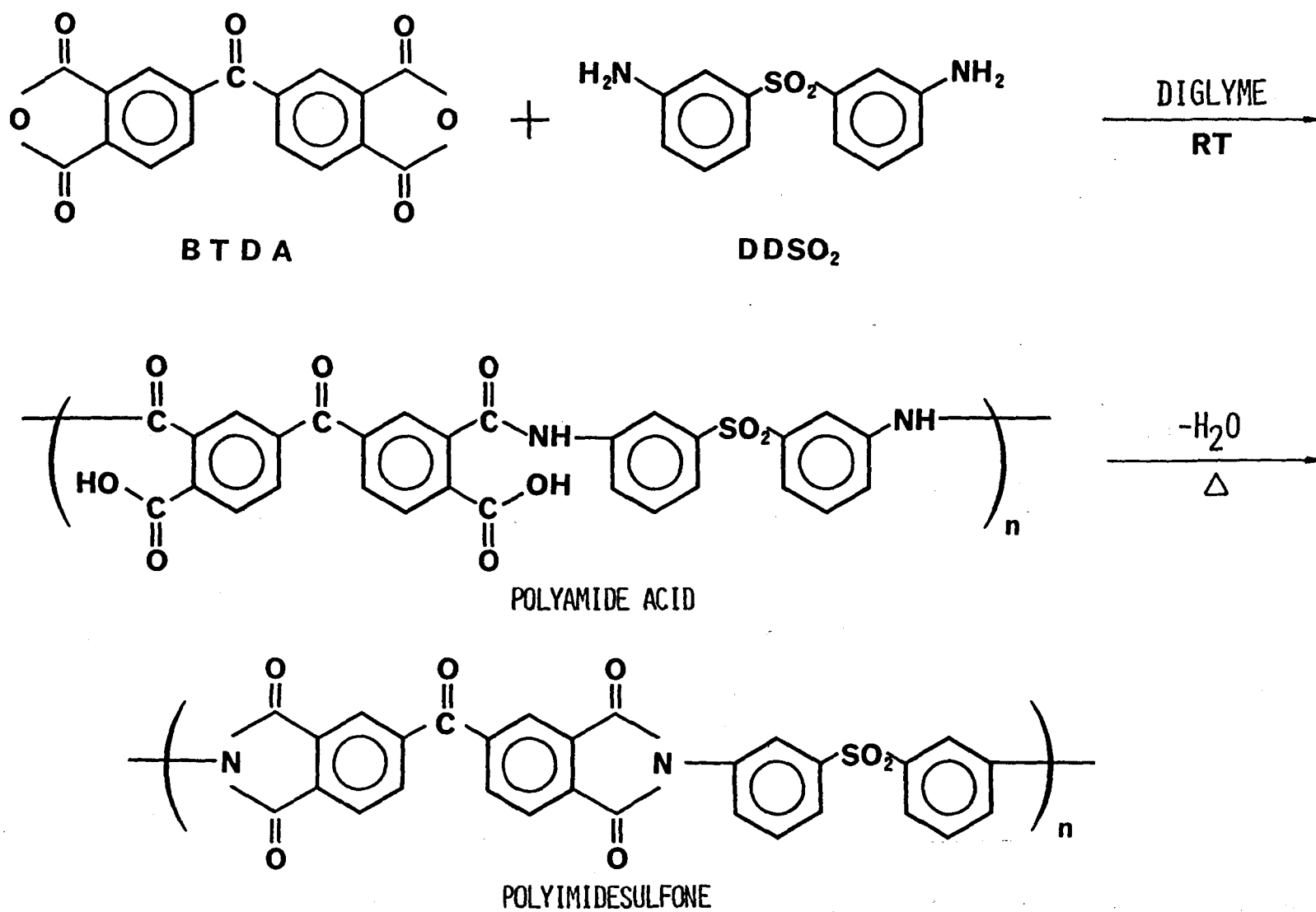


Figure 6. Reaction scheme of polyimidesulfone adhesive.

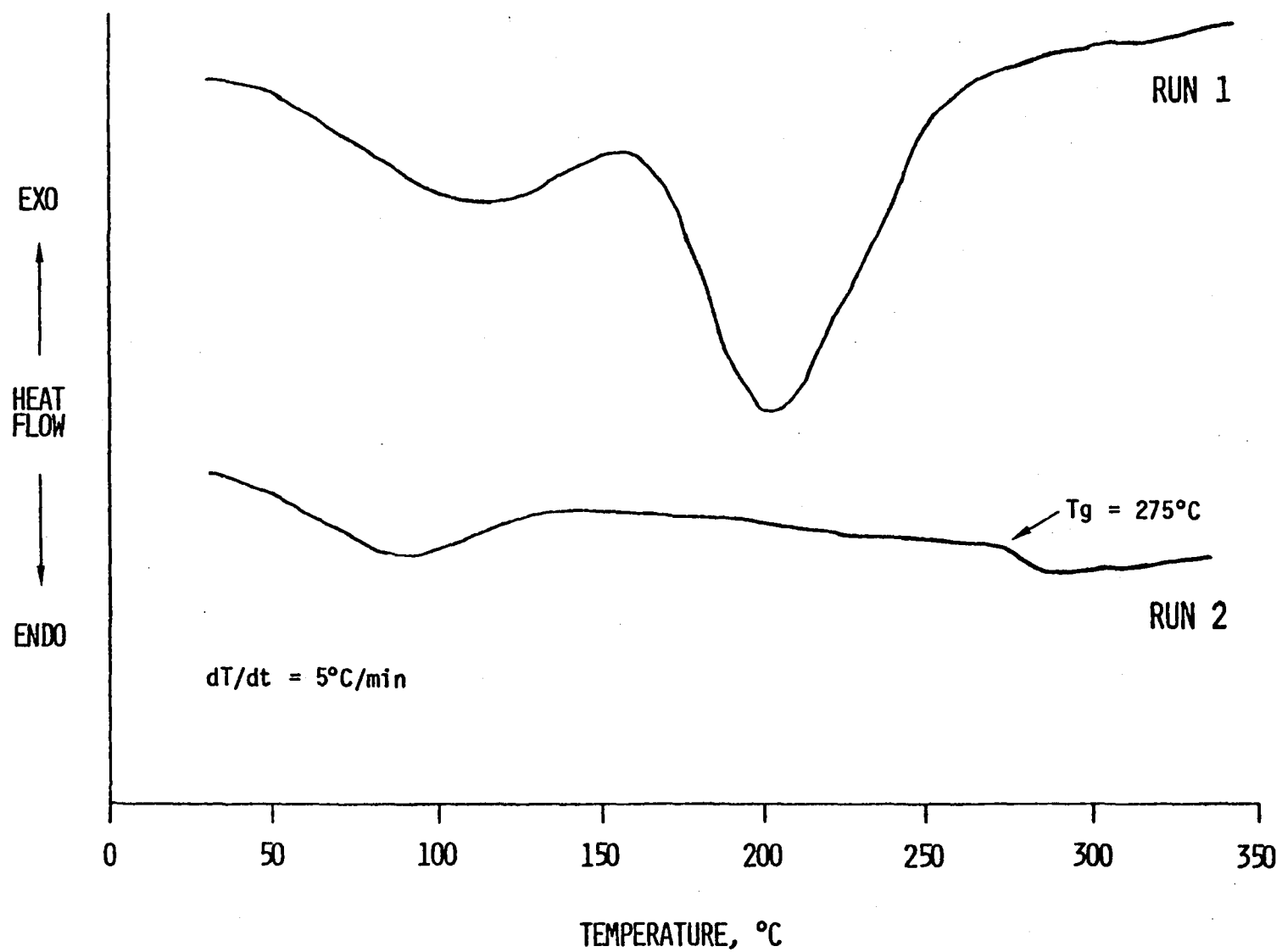


Figure 7. DSC of polyamide-acid powder.

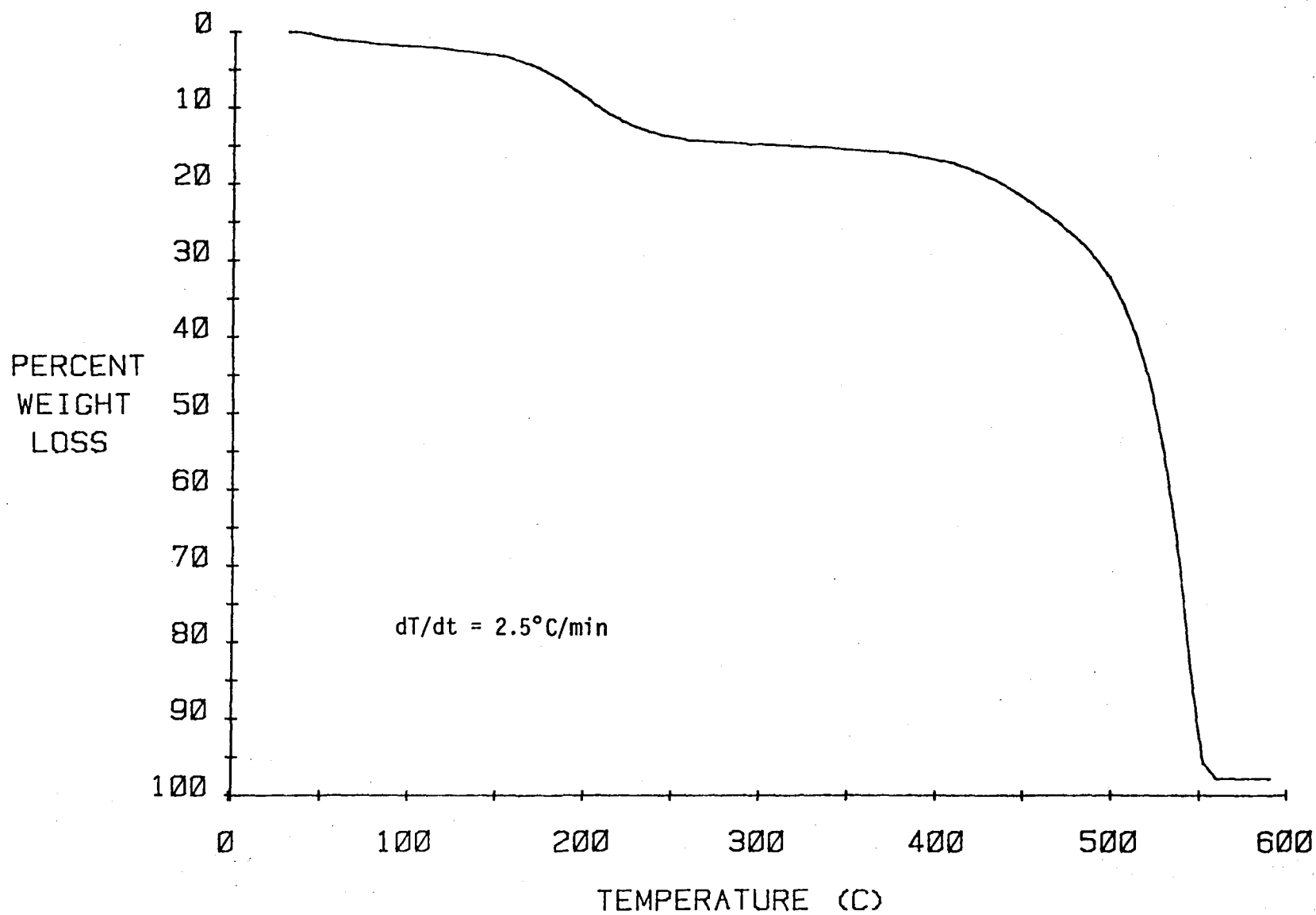


Figure 8. TGA of polyamide-acid powder.

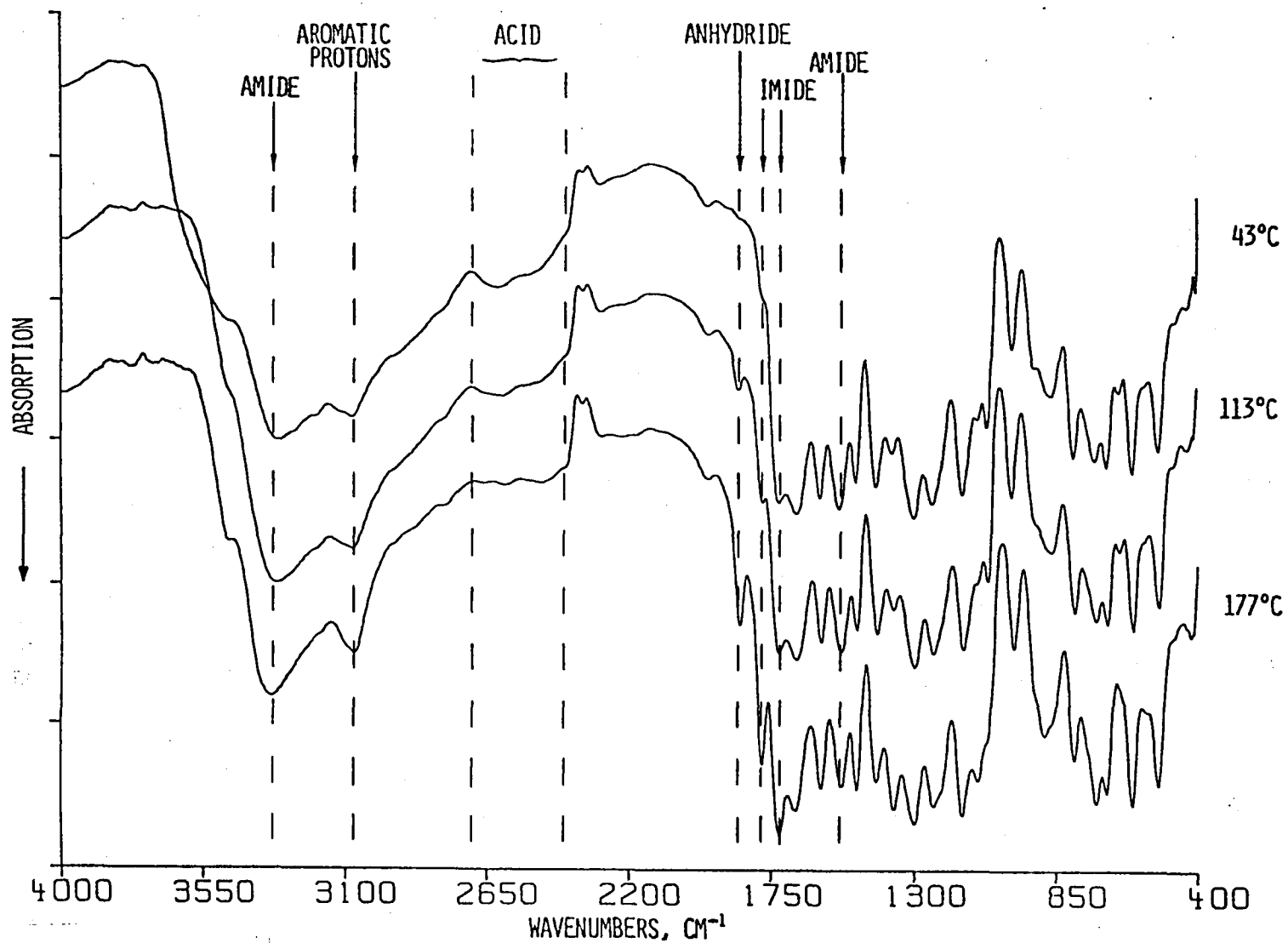


Figure 9. DR-FTIR spectra of polyamide-acid powder during imidization.

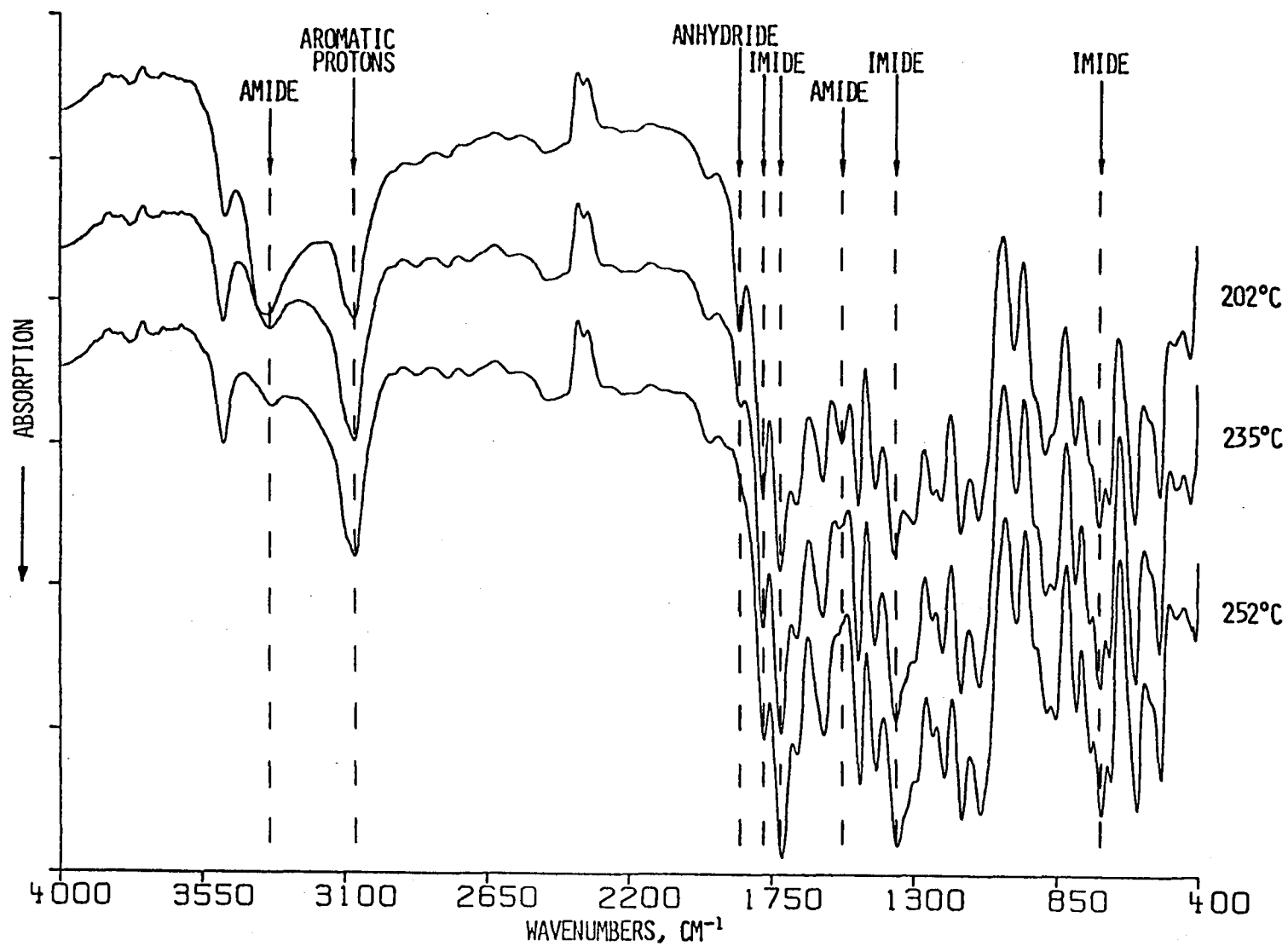


Figure 10. Continuation of DR-FTIR spectra of polyamide-acid powder during imidization.

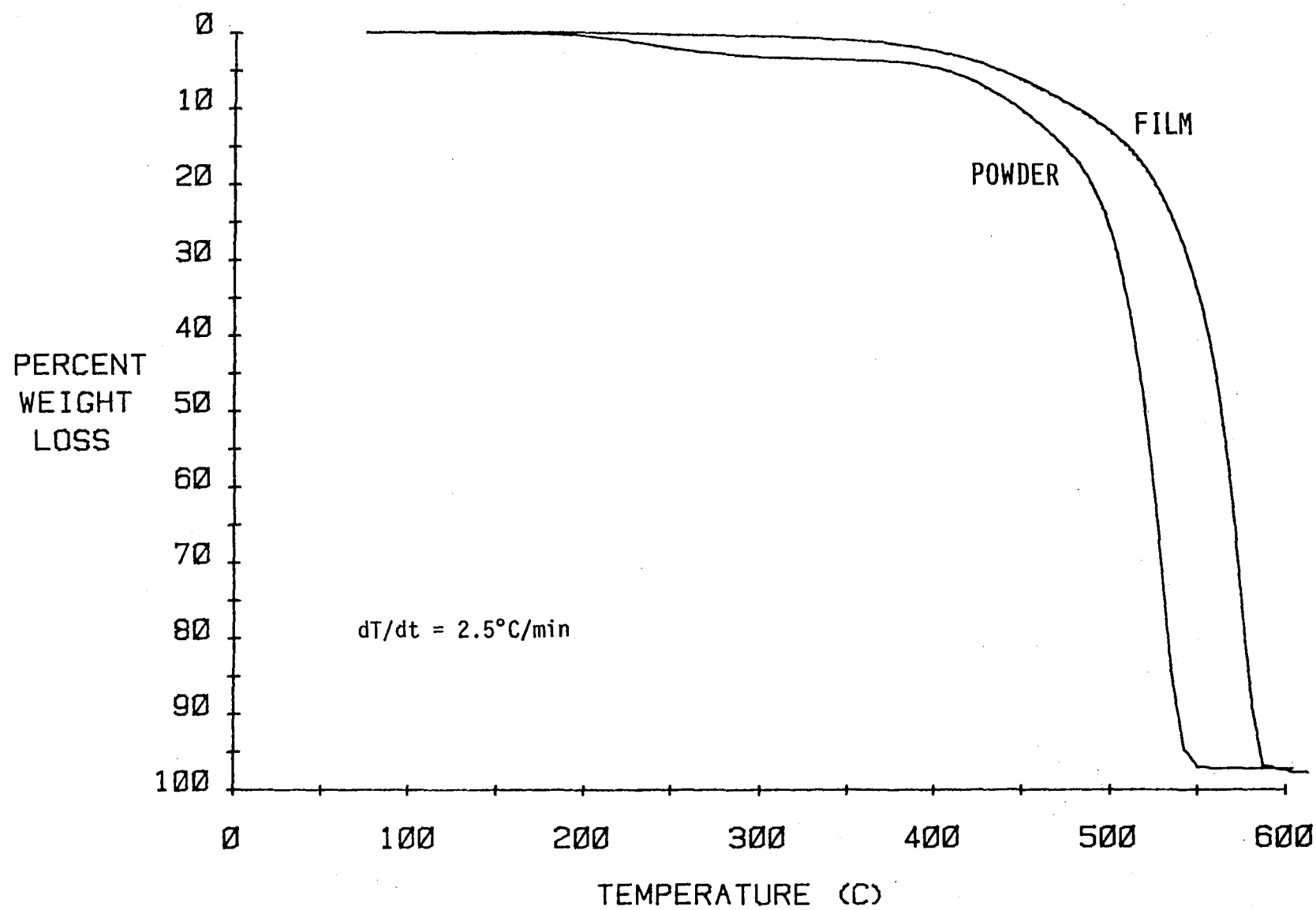


Figure 11. TGA of PISO₂ powder and film after 5-day cure at 150°C.

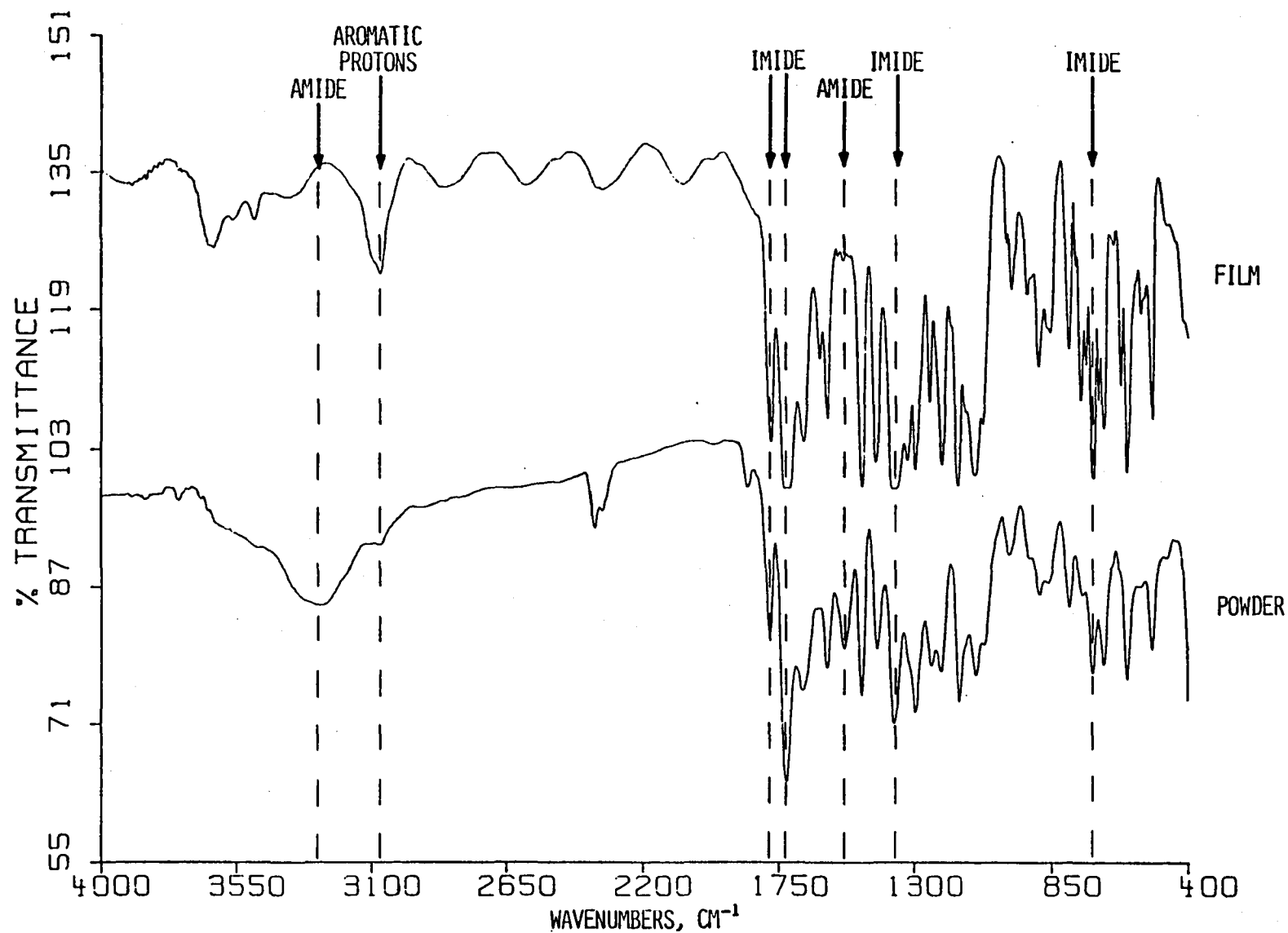


Figure 12. Infrared spectra of PISO₂ powder and film after 5-day cure at 150°C.

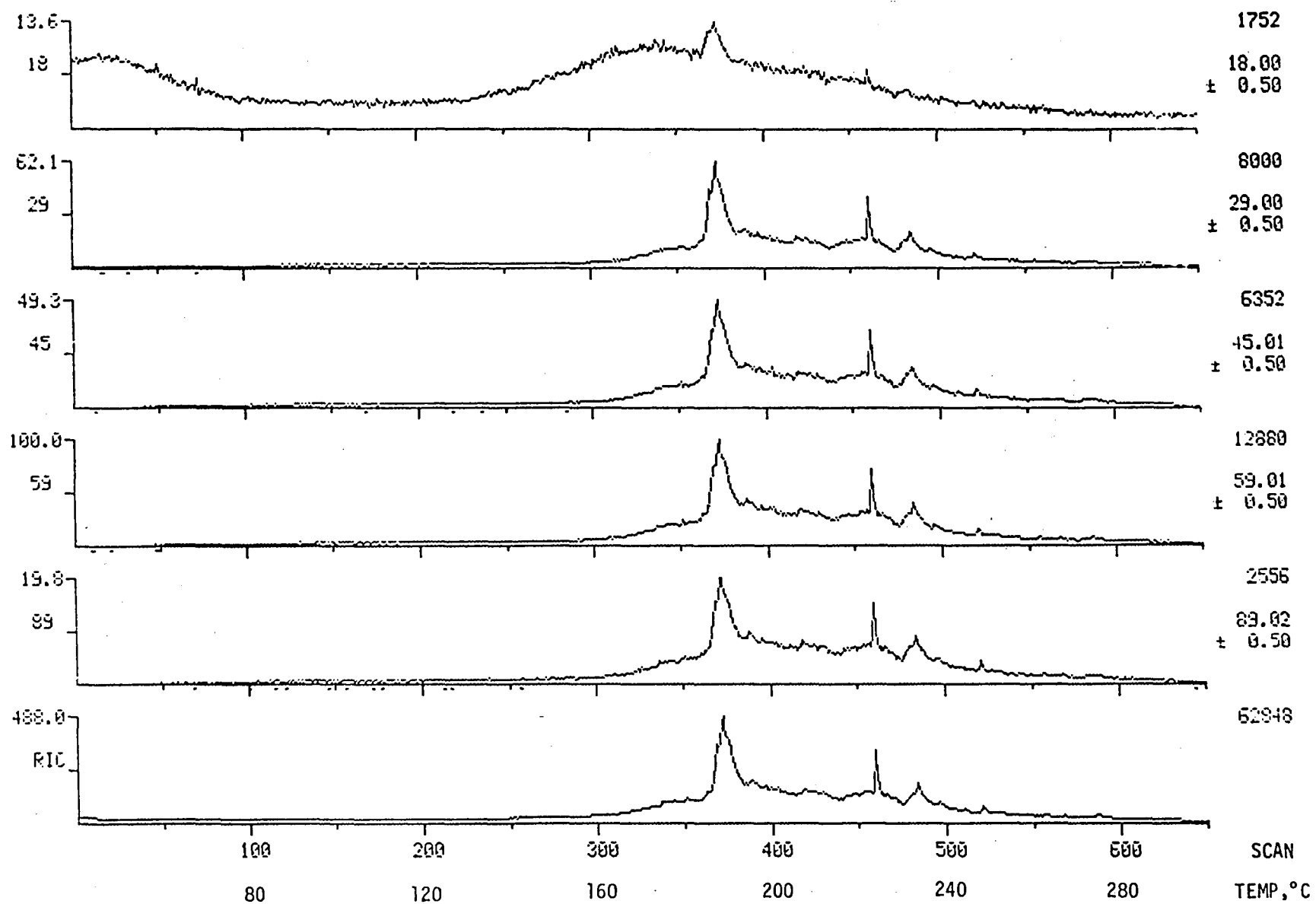


Figure 13. Mass and ion chromatograms of PISO₂ adhesive scrim cloth.

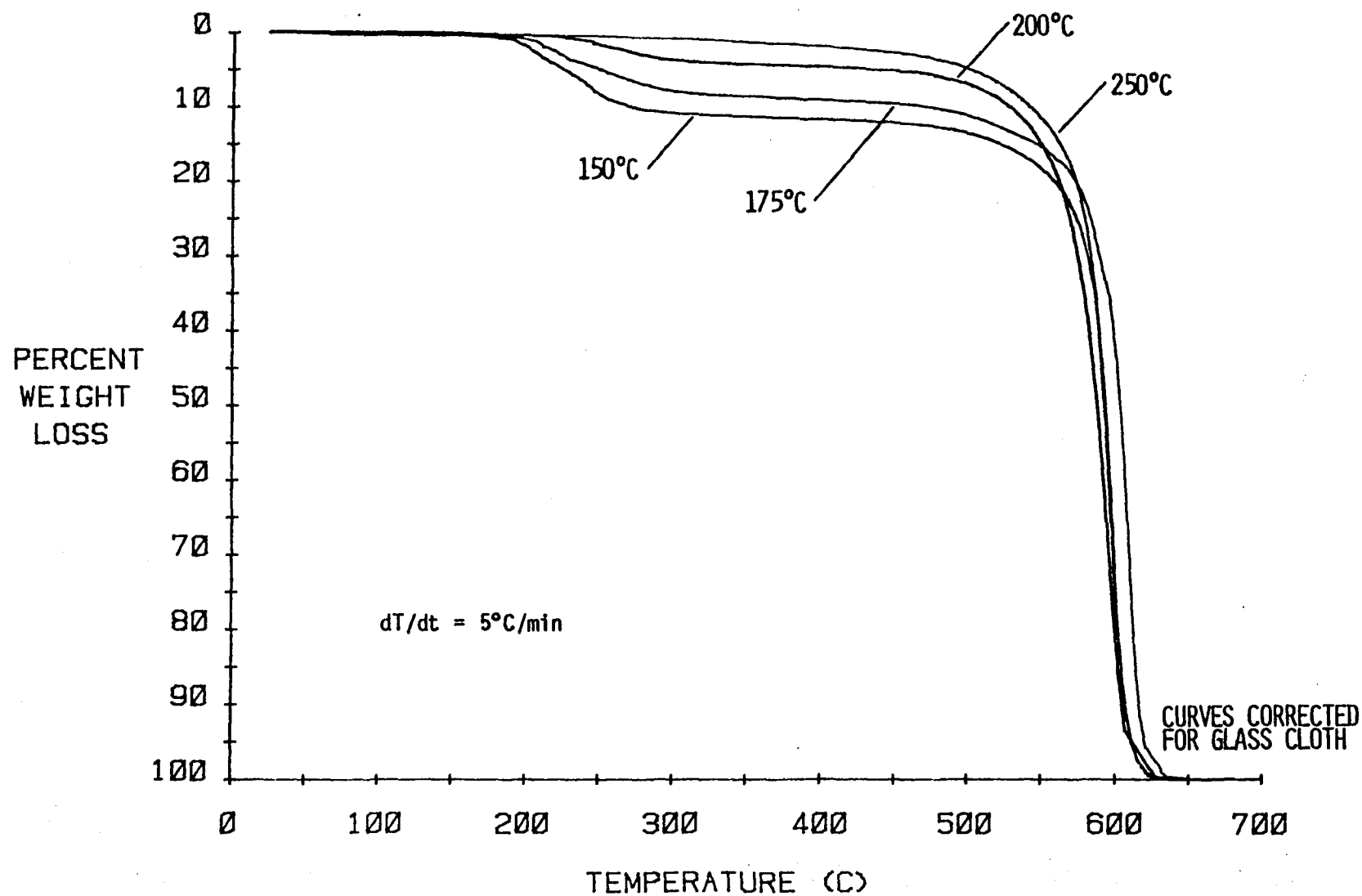
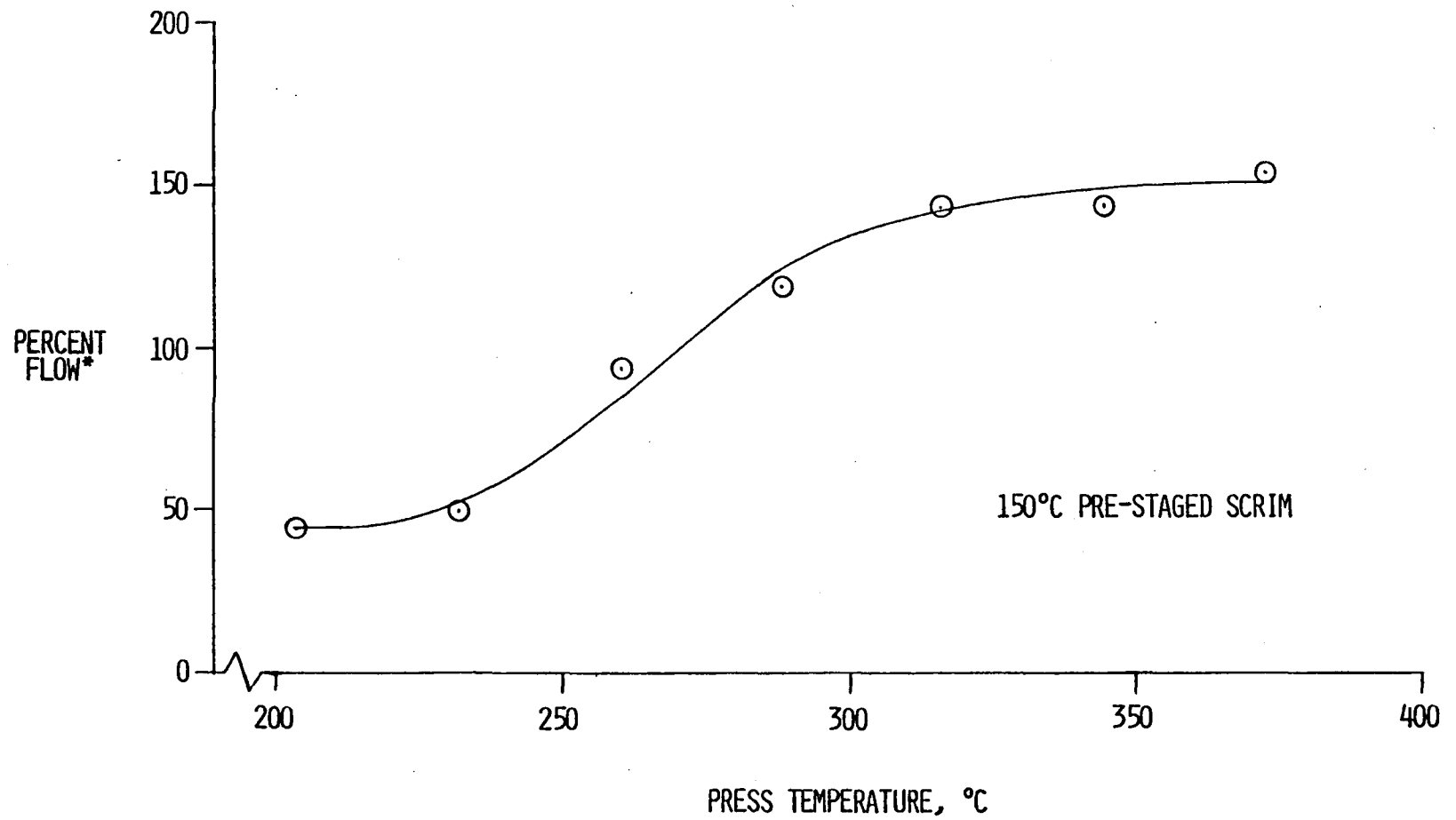
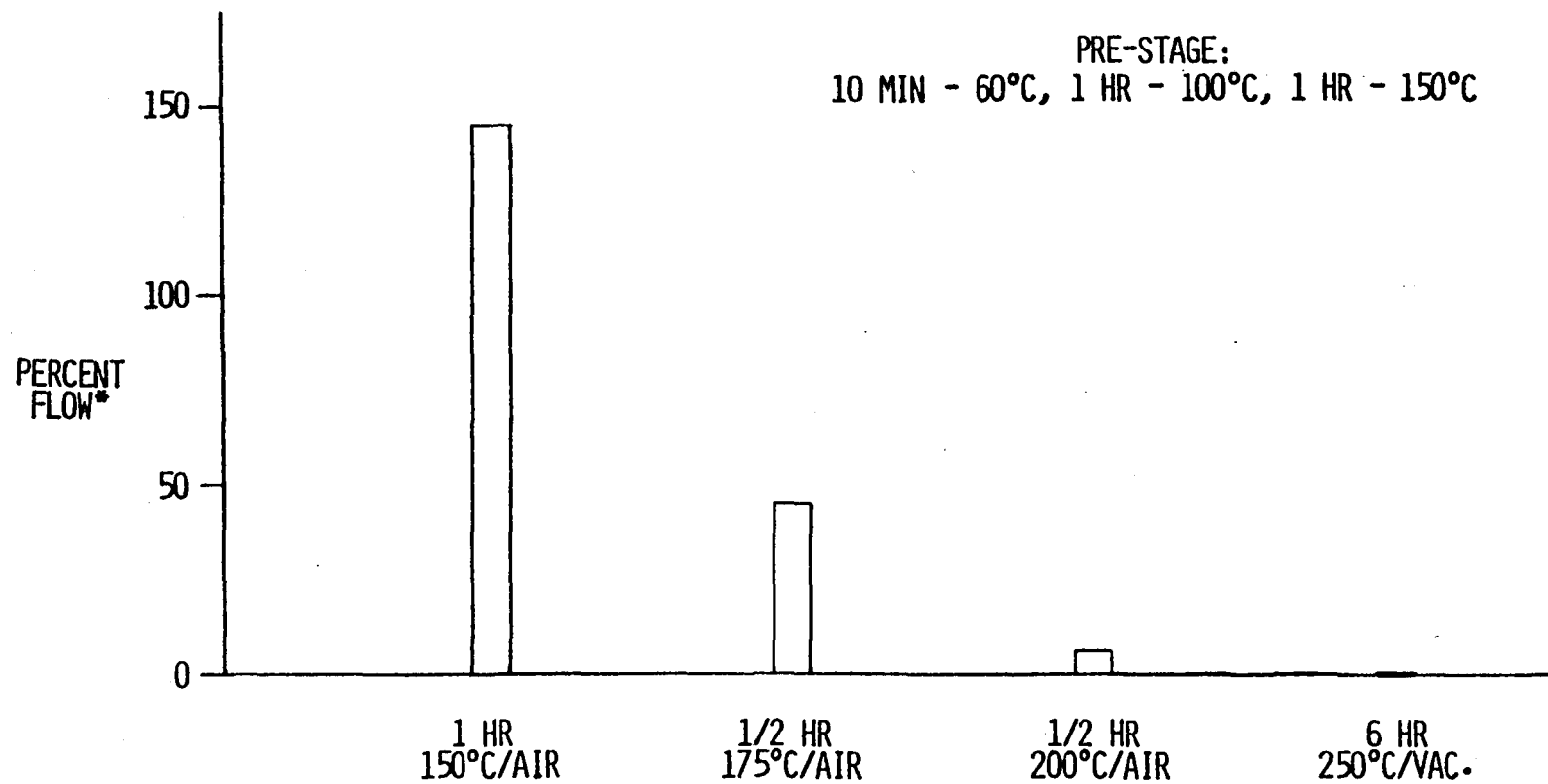


Figure 14. TGA curves of pretreated PISO₂ adhesive scrim cloth.



*FLOW MEASURED UNDER CONSTANT PRESSURE (200 PSI) AT INDICATED TEMPERATURE FOR 3 MINUTES

Figure 15. Flow measurements of PISO_2 adhesive scrim cloth.



*FLOW MEASURED AT CONSTANT PRESSURE (200 PSI) AND TEMPERATURE (343°C) FOR 3 MINUTES

Figure 16. Effect of adhesive scrim cloth pretreatment on flow characteristics.

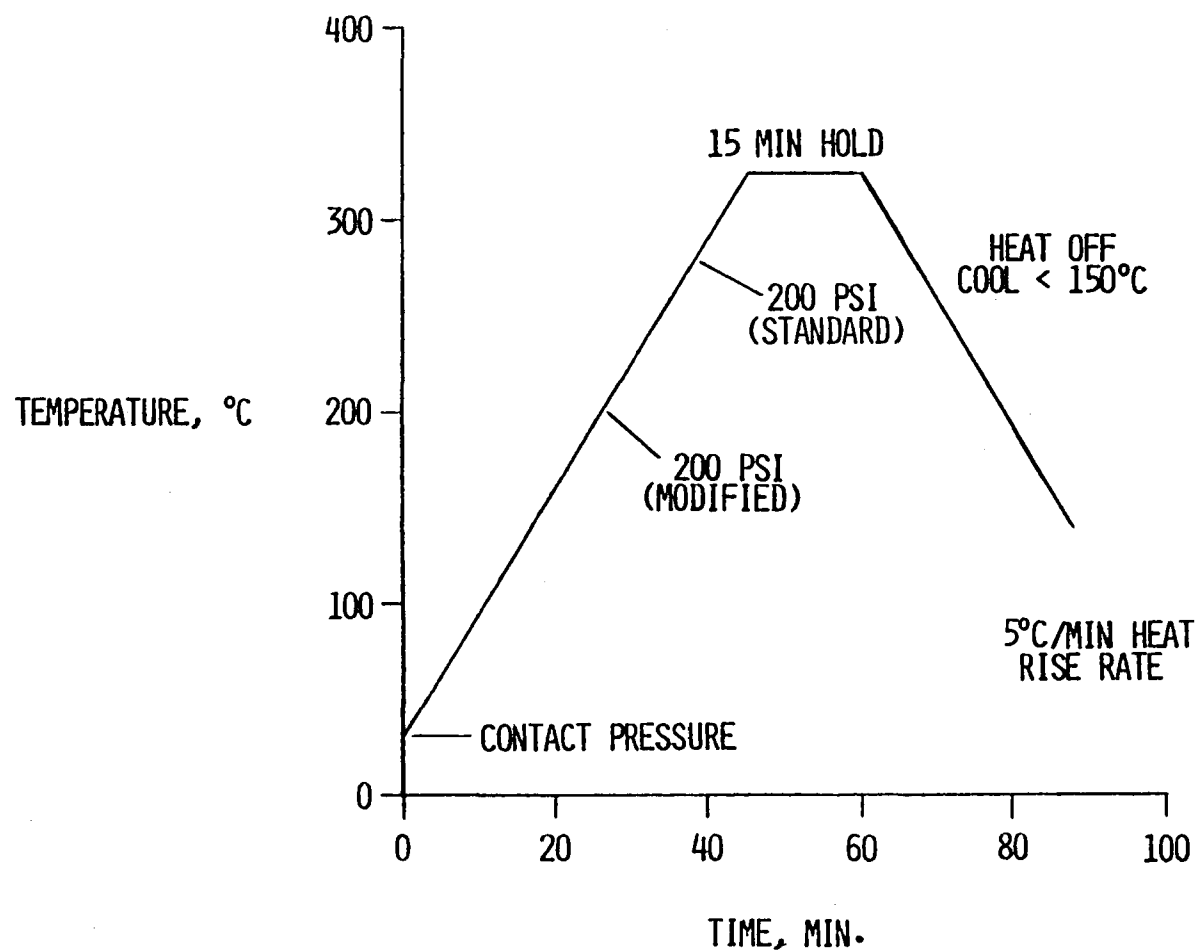
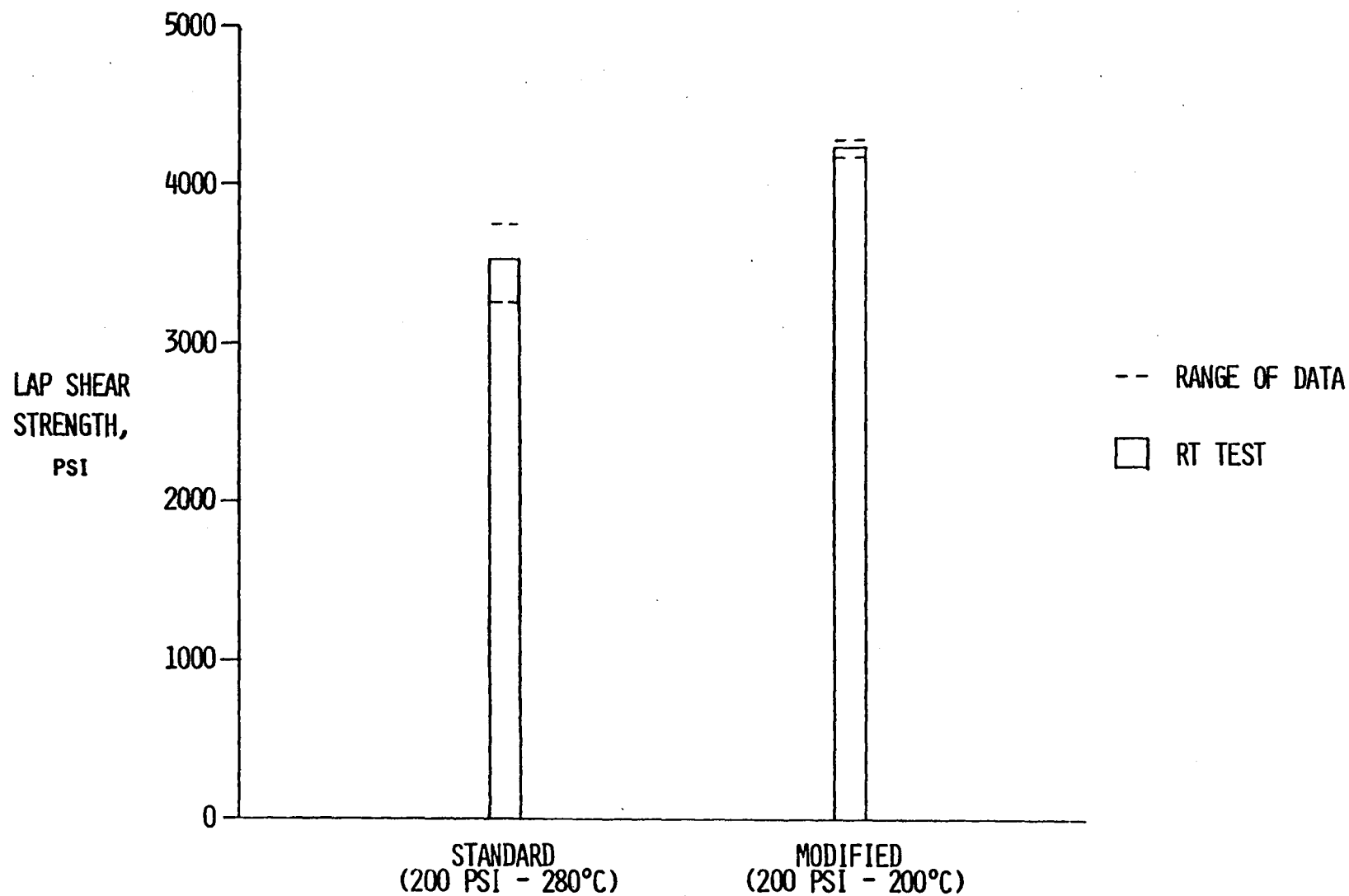
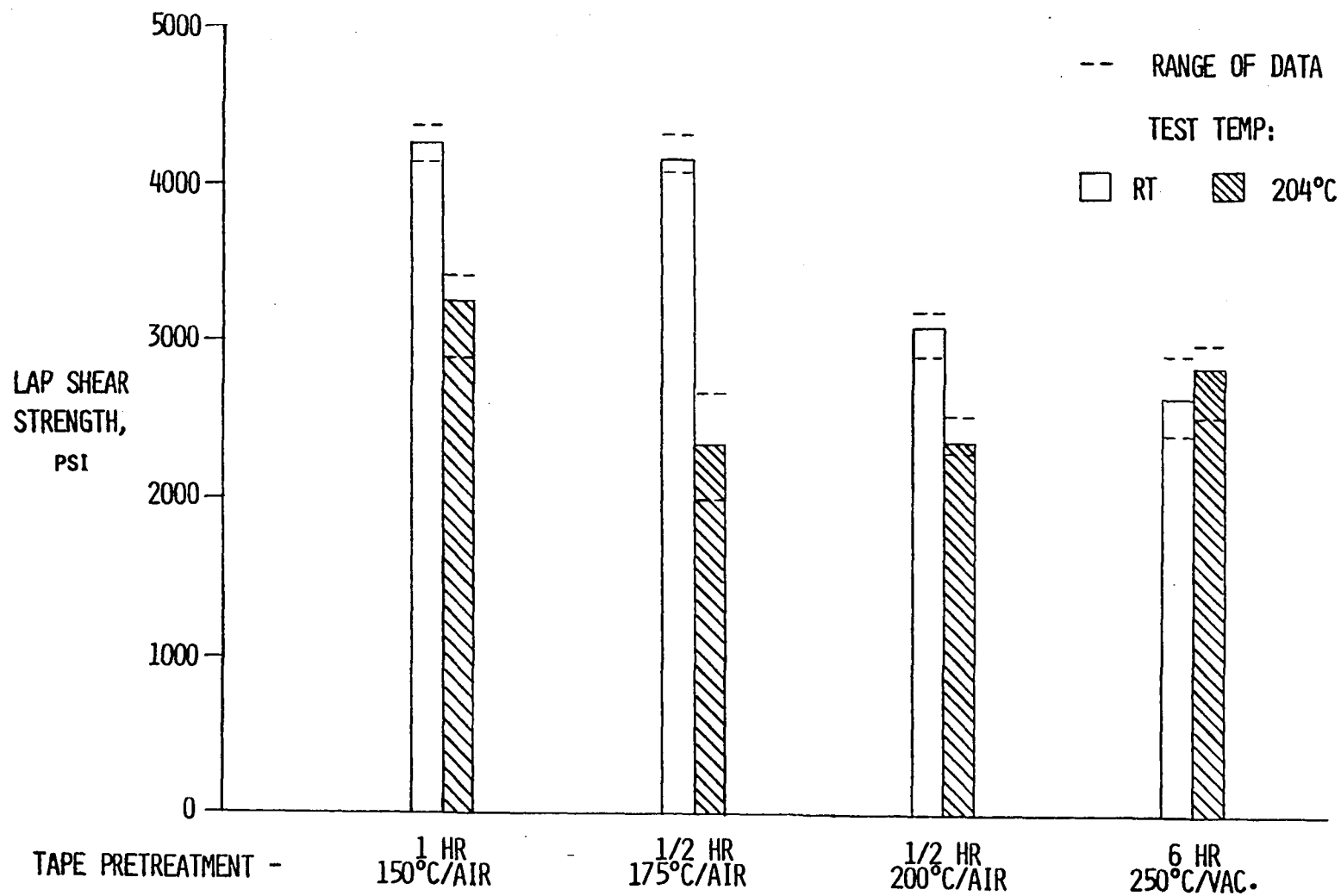


Figure 17. Polyimidesulfone processing cycles.



BONDING CYCLE: RT → 325°C (5°C/MIN), HOLD 15 MIN;
USING PRIMED Ti 6Al-4V ADHERENDS

Figure 18. Lap shear strengths of standard and modified cycles for 150°C pretreated specimens.



BONDING CYCLE: RT + 325°C (5°C/MIN), HOLD 15 MIN, APPLY 200 PSI AT 200°C; USING PRIMED Ti 6Al-4V ADHERENDS

Figure 19. Lap shear strengths of modified cycle for pretreated adhesive specimens.

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16. Abstract This study reports the detailed characterization of an experimental thermoplastic polyimidesulfone adhesive based on 3,3'-diaminodiphenylsulfone and 3,3',4,4'-benzophenone tetracarboxylic dianhydride. Model compounds were also examined. Thermal cyclization of the amide-acid to the imide was studied by a variety of techniques including DSC, TGA, MS, in situ diffuse reflectance-FTIR, and flow measurement. Characterizations were continued during the processing of adhesive tapes and the fabrication, bonding, and testing of lap shear specimens. Results provide fundamental insights into the role of cure chemistry, and the effects of residual solvent and volatile produces on processing and performance. These insights and the resulting chemical models should lead to more efficient processing cycles for these and other related thermoplastic adhesive systems.					
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